IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
(Case No. 97,067)

In re Application of:)	
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F. Leo Hickey)	
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Serial No.: 08/949,239)	Examiner: R. Gorr
)	
Filed: October 10, 1997)	Group Art Unit: 1711
)	
For: POLYOL BLENDS FOR PRODUCING)	
HYDROCARBON-BLOWN)	
POLYURETHANE AND)	
POLYISOCYANURATE FOAMS)	

DECLARATION OF F. LEO HICKEY

Commissioner for Patents and Trademarks
Washington, D.C. 20231

Sir:

I, F. Leo Hickey, of 215 S. Chestnut Avenue, Arlington Heights, IL 60005, declare as follows:

1. I received a B. Sc. degree in Chemistry from Lehigh University, Bethlehem, PA, in 1968.

2. I presently am employed as a Senior Research Chemist at Stepan Co., Northfield, IL.

3.   From 1964 until the present time, I have been employed in various research positions at Stepan Co., Northfield, IL.

4.   I am the author or co-author of two papers, one related to agricultural chemicals, and the other related to polyurethanes.

5.   Because of my extensive background in emulsifiers, surfactants, and polyols, I consider myself an expert in the area of polyols and their use in forming polyurethane and polyisocyanurate foams. I am inventor of this application and submit this declaration in support of the patentability of my invention.

6.   I reviewed the Examiner's Answer dated November 13, 1998 and the pending claims and note that the Examiner had again rejected the claims under 35 U.S.C. § 103(a) as being unpatentable over U.S. Patent No. 4,644,048, Magnus et al. ("Magnus") in view of van der Wouden, "The Use of Polyester Polyols in CFC-Free Rigid Foams," Utech '94, Paper 21, pp. 1-5 ("van der Wouden (Utech 94)"). I submit that Magnus in view of van der Wouden (Utech 94) does not suggest to an ordinary skilled artisan a reasonable expectation of success in making the compositions of the instant claims.

7.   Van der Wouden (Utech 94) teaches that low polarity polyols are needed for hydrocarbon compatibility. Applicant's response dated October 10, 1998 argued that the polyols of Magnus would have higher polarities and thus would not be expected to demonstrate hydrocarbon compatibility. The Office Action dated November 13, 1998 concluded that the hydrophobic component of Magnus would lower the polarity sufficiently to enable compatibility with the hydrocarbon blowing agent.

8.   In van der Wouden (Utech 94), three types of dimer acid-based polyester polyols were added to a hydrocarbon-incompatible polyether polyol. At constant polyether/polyester ratio, hydrocarbon compatibility increased as polyester polyol polarity decreased.

9.   Van der Wouden (Utech 94) identifies the polyester polyol enabling best hydrocarbon solubility as dimer acid-based "Unichema polyester OHV 345." *See* page 2, column 1, line 52. Unichema sells this polyester polyol, which has a hydroxyl value ("OHV") of 345, under the tradename Priplast 3185. *See* van der Wouden (Utech Asia 95), page 2, column 1, line 54. Van der Wouden, "Performance of Oleochemical Based Polyester Polyols in Polyurethanes," Utech Asia '95, Paper 34, pp. 1-7 ("van der Wouden (Utech Asia 95)") at pages 2-3 identifies Priplast 3181 as a polyester polyol having hydrocarbon compatibility even better than that of Priplast 3185.

10.   Although neither van der Wouden (Utech 94) nor Unichema identifies how much of the hydrophobic dimer acid is present in the polyester polyols which provide hydrocarbon compatibility, one of ordinary skill in the art can estimate the dimer acid content of a given polyester polyol using various methods.

11.   Van der Wouden (Utech 94) characterizes the Unichema polyester polyols studied as being based on a dimer acid esterified "with polyalcohols." *See* van der Wouden (Utech 94), page 2, column 1, line 1. Van der Wouden (Utech Asia 95) characterizes the investigated Unichema polyester polyols as being derived from transesterification of dimer acid "with polyols such as glycerine." *See* van der Wouden (Utech Asia 95), page 1, column 2, line 25. Therefore, for the purpose of estimating dimer acid content, it would be reasonable for one familiar with polyester polyols to assume Priplast 3181 and Priplast 3185 are reaction products of dimer acid and glycerol.

12.   One method of estimating dimer acid content is by examining the relative densities of the reactants and the product. The density of glycerol is 1.261 g/ml. *See* Aldrich Chem. Co. catalog, 1996-97, page 759. The Unichema publications list the density of dimer acid (Pripol 1008), polyester polyol Priplast 3185, and polyester polyol Priplast 3181 as 0.95 g/ml, 1.05 g/ml, 1.07 g/ml, respectively. *See* High Purity Dimer Fatty Acids In Condensation Polymers, Unichema Int'l, October 1989; Priplast Polyester Polyols for the Polyurethane Industry, Unichema Int'l, no date available; Priplast Polyester Polyols for the

Polyurethane Industry, Unichema Int'l, May 1995. Because there is a considerable difference between the densities of the two reactants (dimer acid and glycerol), and the only loss on reaction is water (a relatively small molecule), one of ordinary skill would reasonably assume that the density of the reaction product can be approximated by the sum of the weight-averaged densities of the reactants, as follows:

reaction product density = (weight % glycerol x density of glycerol) +

(weight % dimer acid x density of dimer acid).

Conversely, once the density of the reaction product is known, the approximate weight percentages of the reactants can be calculated. For the Priplast 3185 of van der Wouden (Utech 94), the above calculation produces the following estimate of dimer acid content: (1.261-1.05)/(1.261-0.95) = 0.68, or 68% dimer acid. For the Priplast 3181 of van der Wouden (Utech Asia 95), the corresponding calculation is: (1.261-1.07)/(1.261-0.95) = 0.61, or 61% dimer acid.

13. An alternative method for estimating dimer acid content involves examining both the stoichiometry of the reaction that produces the polyol and the properties of the reaction product polyol. If one mole of dimer acid (Pripol 1008, average acid value 196, equivalent weight 286, molecular weight 572) is fully esterified with 2 moles of glycerol (molecular weight 92) to generate a polyol, the dimer acid comprises 76 percent by weight based on the total weight of reactants. However, the expected hydroxyl value of such a reaction product is only 312, compared to quoted average hydroxyl values of 345 for Priplast 3185 and 445 for Priplast 3181. If enough excess glycerol (hydroxyl value 1827) is used in the reaction to bring the final hydroxyl values to 345 and 445, respectively, the compositions are:

For Priplast 3181:

8.8% by wt. free glycerol

91.2% by wt. dimer acid/glycerol ester

For Priplast 3185:

> 2.2% by wt. free glycerol
>
> 97.8% by wt. dimer acid/glycerol ester

These final compositions would theoretically require the following amounts of starting materials:

For Priplast 3181:

> 572 parts by wt. dimer acid
>
> 184 parts by wt. glycerol to enter into the esterification
>
> 70 parts by wt. free glycerol

For Priplast 3185:

> 572 parts by wt. dimer acid
>
> 184 parts by wt. glycerol to enter into the esterification
>
> 16 parts by wt. free glycerol

The percentages of dimer acids in these mixtures are:

> Priplast 3181: <u>69% dimer acid</u>
>
> Priplast 3185: <u>74% dimer acid</u>

14.   Both estimation methods suggest that the content of hydrophobic component required for enabling hydrocarbon compatibility is well over 50 percent by weight. Thus, after considering van der Wouden (Utech 94), an ordinary skilled artisan would expect that the amount of hydrophobic soybean oil required to produce hydrocarbon compatibility in an otherwise polar phthalic anhydride-based polyol, like those of Magnus, is also likely to be well over 50 percent by weight.

15. However, one skilled in the art would also be concerned with the ultimate physical properties of the polyurethane foam that could be produced from such a polyester polyol, given the fact that dimer acids are difunctional and soybean oil fatty acids are monofunctional. One skilled in the art is aware that a minimum of two functional groups per molecule is necessary in polyurethane-forming reactions to allow extensive polymer chain development and produce high polymer strength and rigidity. A high content of monofunctional material, on the other hand, will cause widespread chain termination and degrade properties. The requirement for two or more functional groups per molecule is met with dimer acid-based polyols. But one of ordinary skill would be concerned that reacting over 50 percent by weight monofunctional material into a difunctional phthalic-based polyol like that of Magnus would negatively impact polymer properties, making the approach impractical.

16. Potential negative effects of reacting greater than 50 percent by weight monofunctional soybean oil into a difunctional phthalic-based polyol may be clarified by considering the results in terms of molar composition. In the present invention, the phthalic-based polyol, before introduction of the hydrophobic material, meets the desirable minimum functionality of two. However, if over 50 percent by weight of monofunctional soybean oil is transesterified into the original polyol, the difunctional content of the polyol is severely reduced. A representative calculation could be based on 50 percent by weight soybean oil and 50 percent by weight of a 480-hydroxyl value phthalic anhydride-based polyol, to give a polyol with a typical final hydroxyl value of 240. The final functionality of this product after reaction is calculated to be 1.43, equivalent to 57 mole percent monofunctional and 43 mole percent difunctional material. Because the final functionality of such a reaction product is much lower than 2, a person skilled in the art of polyurethane polyols would expect this approach to be unworkable.

17. In view of the fact that the above analysis, which is based on information available at the time of the invention, indicates that the van der Wouden (Utech 94)

approach requires excessively high levels of reacted hydrophobic materials, it is surprising to find that much lower levels of these materials (in combination with surfactants) are effective in compatibilizing high percentages of hydrocarbons. The ability of the claimed invention to use lower levels of monofunctional hydrophobic residues avoids the problem of excessively reducing functionality and the expected possibility of resulting inferior polymer properties.

18.   Van der Wouden (Utech 94) teaches that well over 50 percent by weight hydrophobic component is required for enabling hydrocarbon compatibility. Magnus is silent on the issue of hydrocarbon compatibility, and teaches only that incorporating up to 28 mole percent of hydrophobic component into a polyester polyol enables fluorocarbon compatibility. U.S. patent No. 4,644,048, Magnus et al., col. 6, line 31. The most preferred range of hydrophobic compound in Magnus is 5 to 15 mole percent. Magnus, col. 7, line 37. Magnus examples show hydrophobic component being utilized at levels of about 20 percent by weight. Magnus, Table X, col. 22, Example 16 (decyl alcohol used at 19.1 wt%); col. 21, Example 9 (soybean oil used at 20.6 wt%). The present invention enables hydrocarbon compatibility even though it incorporates less than about 40 percent by weight hydrophobic component. An ordinary skilled artisan considering Magnus in view of van der Wouden (Utech 94) would not expect that incorporating only about 40 percent by weight or less of hydrophobic component would sufficiently lower the polarity of the polyester polyol to enable compatibility with a hydrocarbon blowing agent.

19.   I declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true and, further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the U.S. Code and that such willful false statements may jeopardize the validity of this application and any patent issuing thereon.

Respectfully submitted,

Dated: 2/15/99

F. Leo Hickey